



Material property characterization and modelling from FIB/SEM nanoscale 3D imaging

EFUG Meeting 2011 October 3rd - Talence, France

Daniel.Lichau@vsg3d.com

- About VSG and Avizo software
- From FIB/SEM imaging to 3D material properties characterization
- Examples in electronics, metals, fuel cells, rocks
- Introducing Avizo version 7

VSG HQ and R&D centre (A)



Proven track record

- 25+ years of expertise
- Uniquely experienced team
- Successful serving 1000+ first-class customers

Worldwide organization

- USA (Boston, Houston, San Diego)
- France (Bordeaux, Paris)
- Germany (Düsseldorf)
- UK (London)

Distributors channel

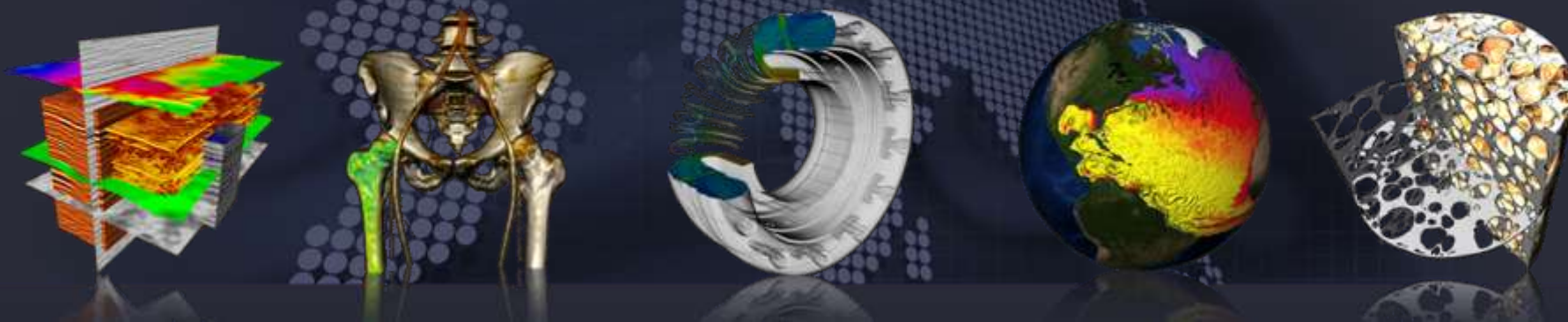
- Asia/Pacific (China, India, Japan, Taiwan, Korea)
- Mexico, Israel, Russia

MARKETS

- Engineering & Simulation
- Oil & Gas, Geosciences, Mining
- Biomedical & Life Sciences
- Materials science, Industrial Inspection
- Scientific data visualization
- Collaborative & Immersive VR

SOLUTIONS

- Object-oriented 3D libraries and API
- Open application framework
- State-of-the-art 3D rendering and analysis
- Very Large Data Management
- Scalability and remote application
- 3D computing framework



Open Inventor®

3D development toolkit



Integrate
high-performance 3D
in your applications

Avizo®

3D application framework

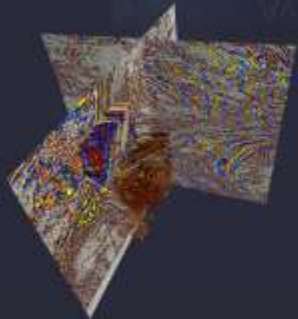
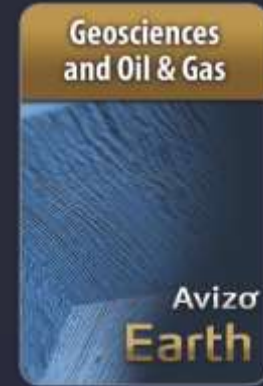


Visualize and
analyze scientific
and industrial data

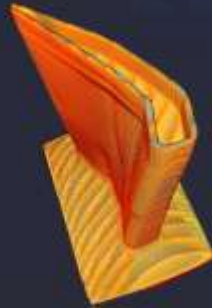
3D expertise and
Professional services



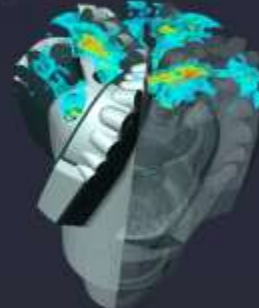
Join a partner
committed to your
success



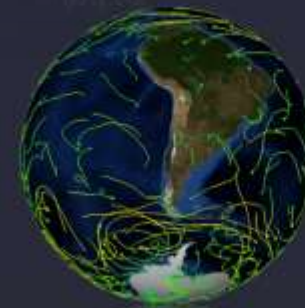
Data: CGG



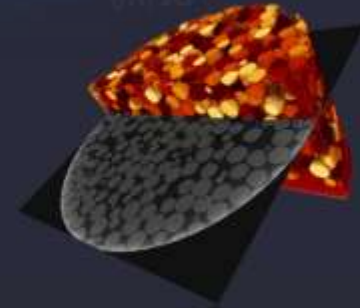
Data: GE Aircraft



Data: VAREL



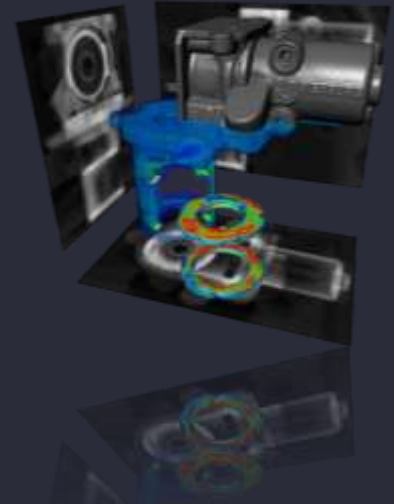
Data: MPI-MP / DRKZ



Data: ICMCB



Avizo **Fire** is a 3D analysis application for Materials Science and Industrial Inspection



Data: North Star Imaging

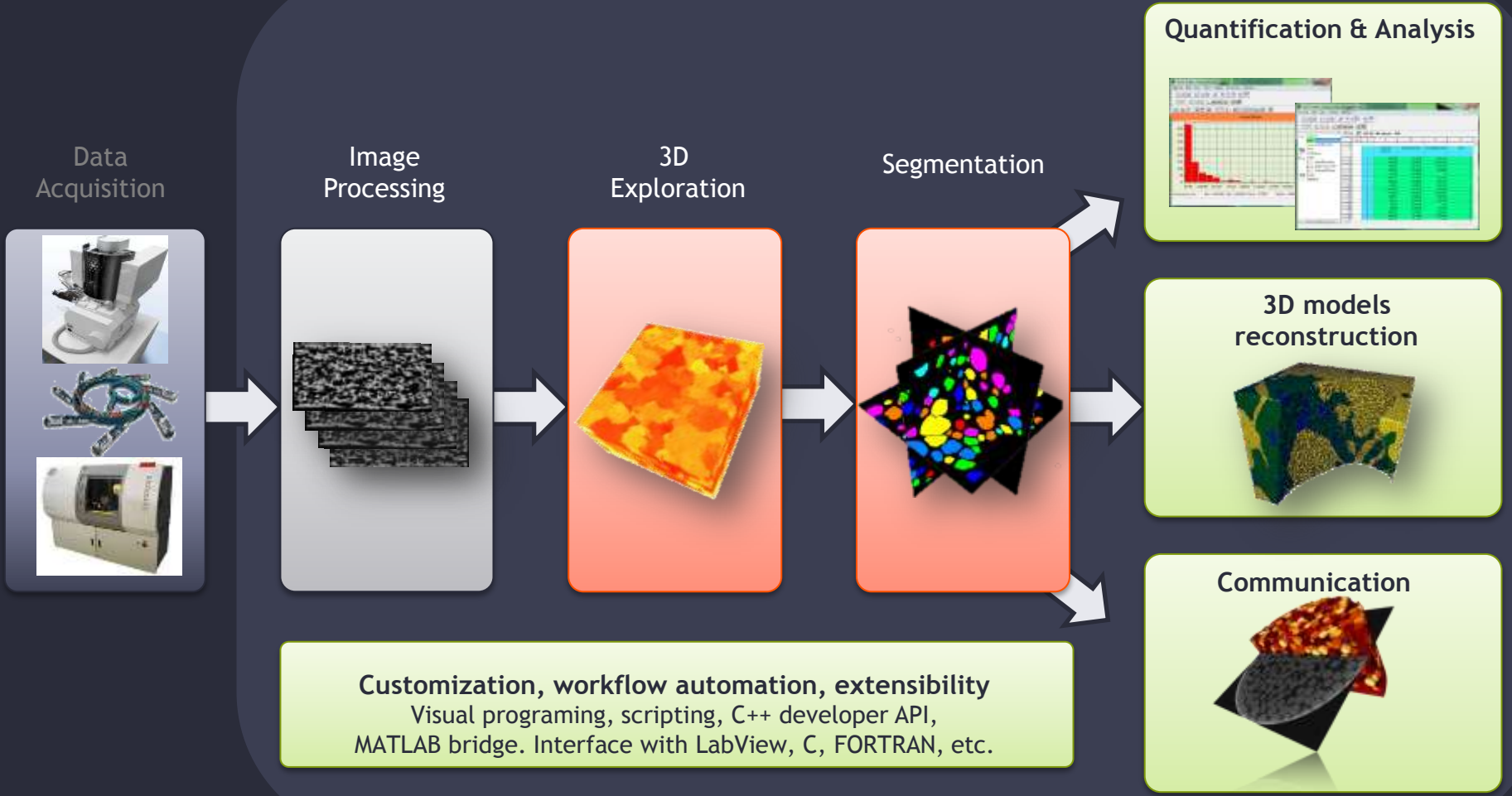
Key Features

- Extensive 3D image import and processing
- Advanced image segmentation
- Quantification and analysis
- High-quality presentation and reporting

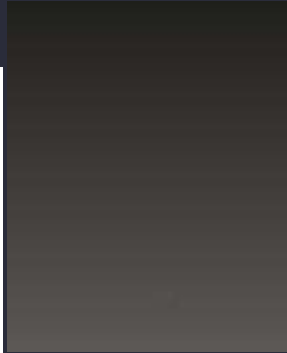
Market applications

- Materials science, micro-CT
- Industrial tomography, Non destructive inspection
- Reverse engineering
- Core sample analysis

Avizo Fire for Materials Science 3D imaging and analysis



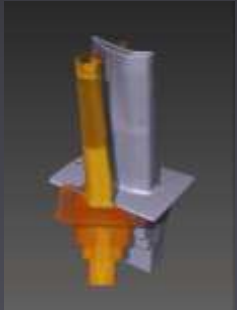
Avizo Automation Framework



INSPECTION DATA SERVICE

Image stack reception

Registration against reference model



Data: GE Aviation

DESIGNER TOOL

Create your inspection scenario(s)

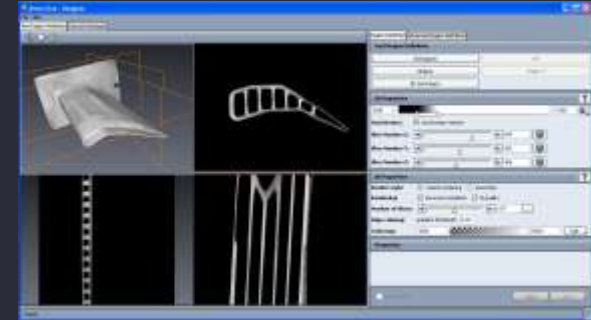
INSPECTOR TOOL

Inspection using your test scenario(s)

Compliant part

Noncompliant part

PRODUCTION LINE



INSPECTION REVIEWER TOOL

Detailed test report

Accept / Reject decision

Compliant part

Noncompliant part



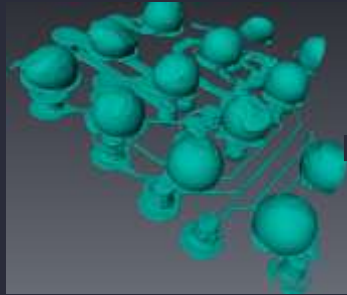
Defect inspection, root cause analysis

Circuit failure,
packaging defect

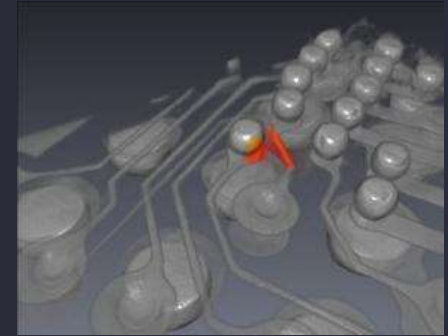
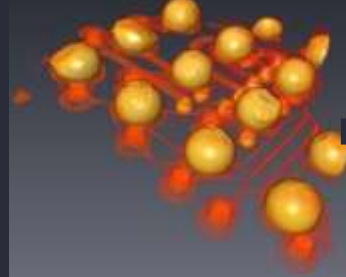


BGA

CT or
FIB/SEM



Advanced
visualization and
data exploration



Defect analysis

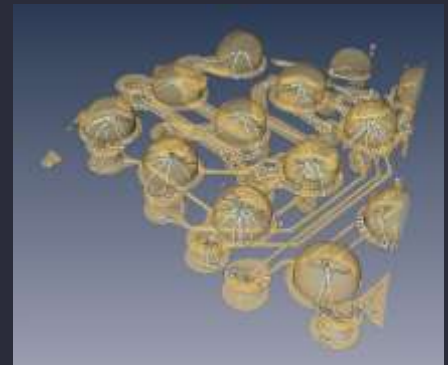
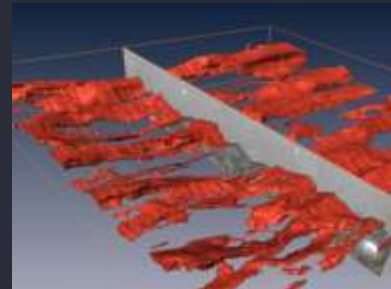
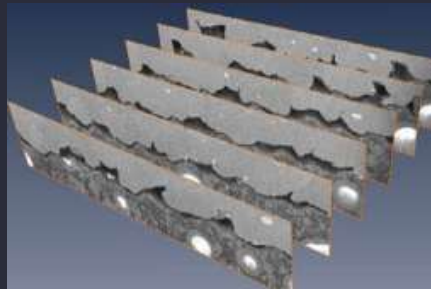
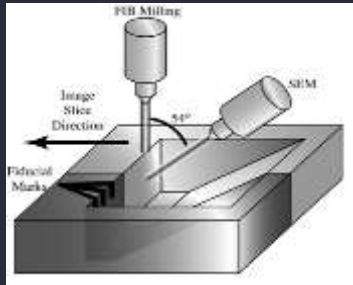
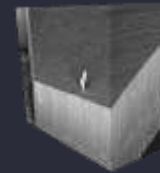
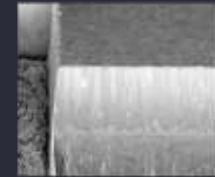
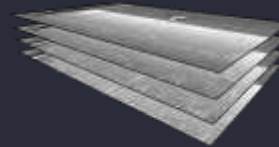


Image skeletonization

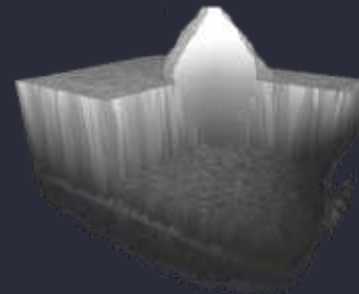
Characterization of delaminations at a chip / molding compound interface
Courtesy A. Rucki

3D FIB-SEM Processing

- Alignment / drift correction
 - Registration (XY)
 - Least square or other methods
 - Optional arbitrary 3D region reference mask
 - Calibration of slice thickness (Z)
- Foreshortening correction (Y)
- Shearing
- Masking/cropping
- Shadow/contrast correction



- Workflow assistant for 3D reconstruction from FIB/SEM images coming in Avizo Fire 7



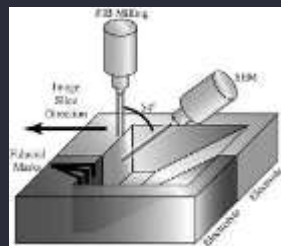
Intermetallic microstructural analysis in tin-plated copper (tin whiskers)

NIST and VSG M. Marsh et al. 2010. *Microscopy and microanalysis*.

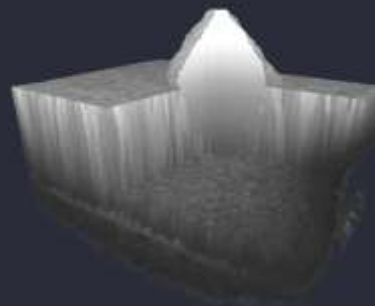
Porous graphitic carbon foam



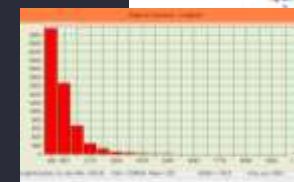
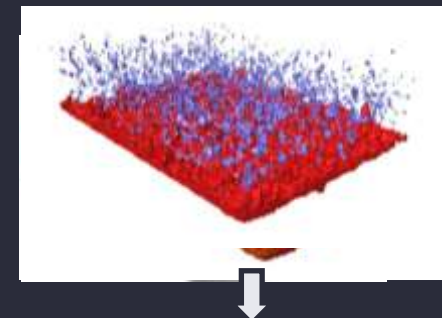
FIB/SEM acquisition



3D Image reconstruction and visualization



3D image segmentation



Quantification and analysis

Images from NASA Goddard Tin Whiskers website

Global metrics, individual measures and population distributions, e.g. :

Fractal dimension of basal surface: **2.31**

Degree of anisotropy of interspersed: **0.598**

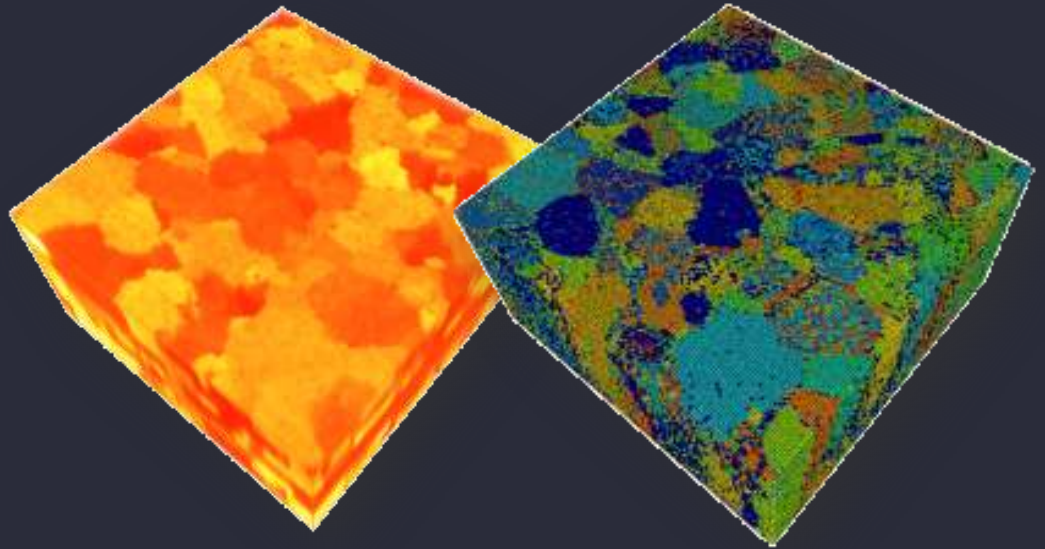
3D Density of interspersed: **0.13 grain / μm^3**

Volume, specific surface, length, width, aspect ratio, orientation, contacts, etc.

3D FIB/SEM images registration and fusion

- Multiple data channels, e.g. BSE, EBSD, EDS, etc. for compositional analysis and enhanced segmentation.
- Multiple datasets, modalities: micro/nanoCT, AFM, etc. for multi-scale and correlative analysis, or for experiment imaging

Example: Multi-channel imaging of microcrystalline metals (IN100 Nickel-based superalloy)
Secondary electron (SE) and electron backscatter diffraction (EBSD)

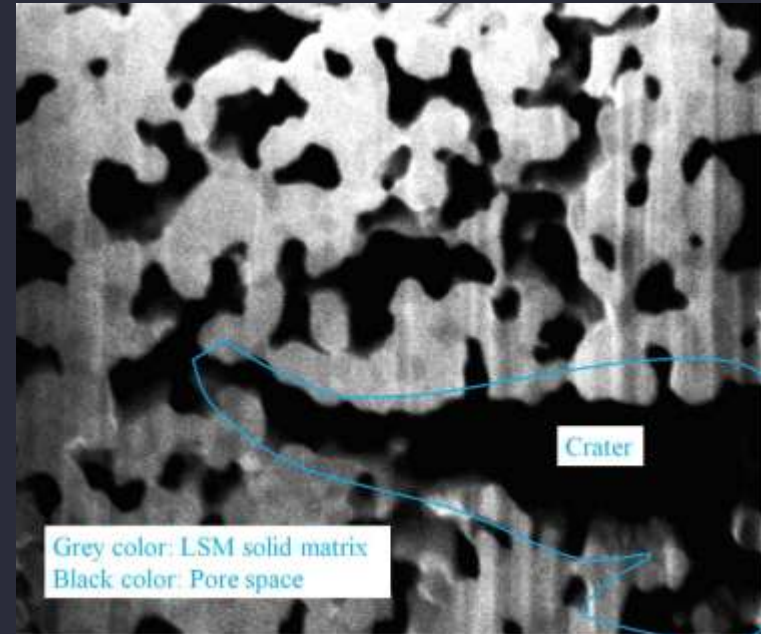


Pilot study of FIB-SEM characterization of microstructure of fuel cells

University of Alberta

Kumar Gunda *et al.* 2011. *J Power Sources*.

- Examine Lanthanum strontium manganite (LSM) matrix Solid oxide fuel cells (SOFCs)
- Filtering and thresholding sufficient to partition matrix from pore-space
- Porosity and surface area sensitive to threshold value

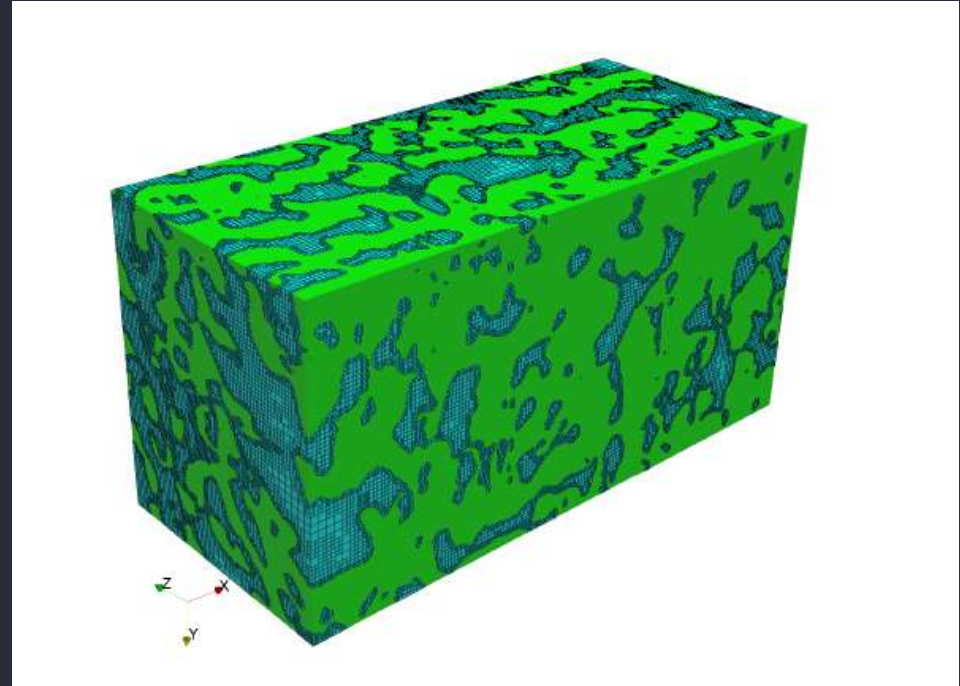


Pilot study of FIB-SEM characterization of microstructure of fuel cells

University of Alberta

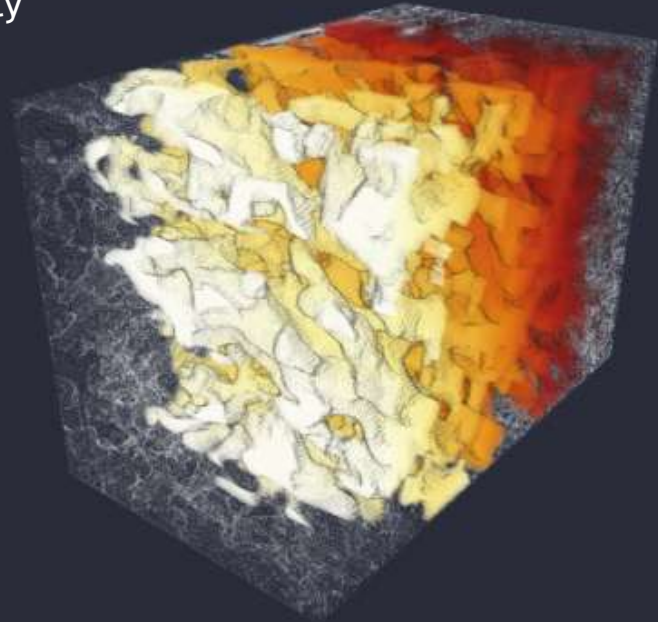
Kumar Gunda *et al.* 2011. *J Power Sources*.

- Examine Lanthanum strontium manganite (LSM) matrix Solid oxide fuel cells (SOFCs)
- Filtering and thresholding sufficient to partition matrix from pore-space
- Porosity and surface area sensitive to threshold value



Characterizing Materials

- Image-based quantification, such as
 - Total porosity, Connected porosity, Included Porosity
 - Specific surfaces
 - Porosity distance, Tortuosity
 - Phases interfaces, multi-phase boundaries, etc.
- Modeling-based quantification, such as
 - Permeability Tensor and Absolute Permeability
 - Molecular Diffusivity
 - Heat Conductivity
 - Formation Factor



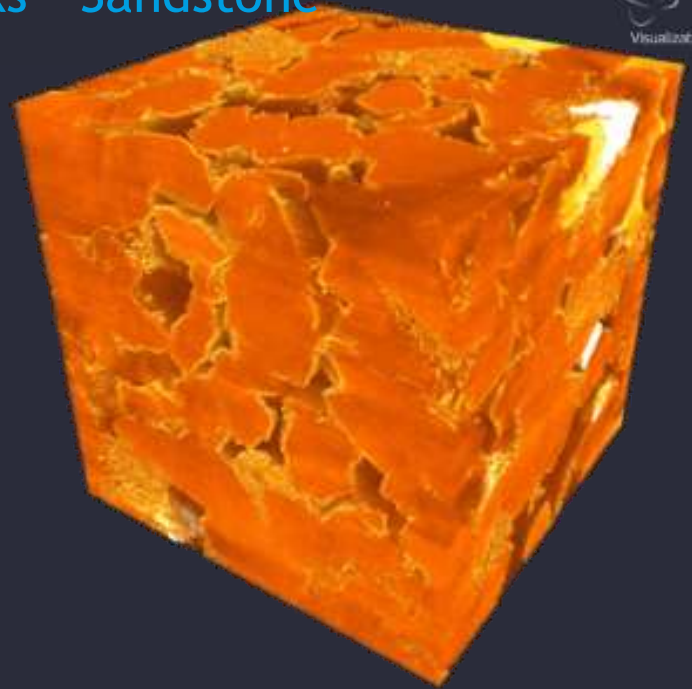
Validation of microCT porosity in berea sandstone

University of Alberta

Bera *et al.* 2011. *Micron*.

Characterizing the pore space in sandstone

- MicroCT
 - 3 μm in-plane, 6 μm slice-thickness
 - Bulk pores (150-200 μm)
 - Small pores (30-80 μm)
 - Suspicious micro-pores ($\sim 5\mu\text{m}$)
near the detection limits



Validation of microCT porosity in berea sandstone

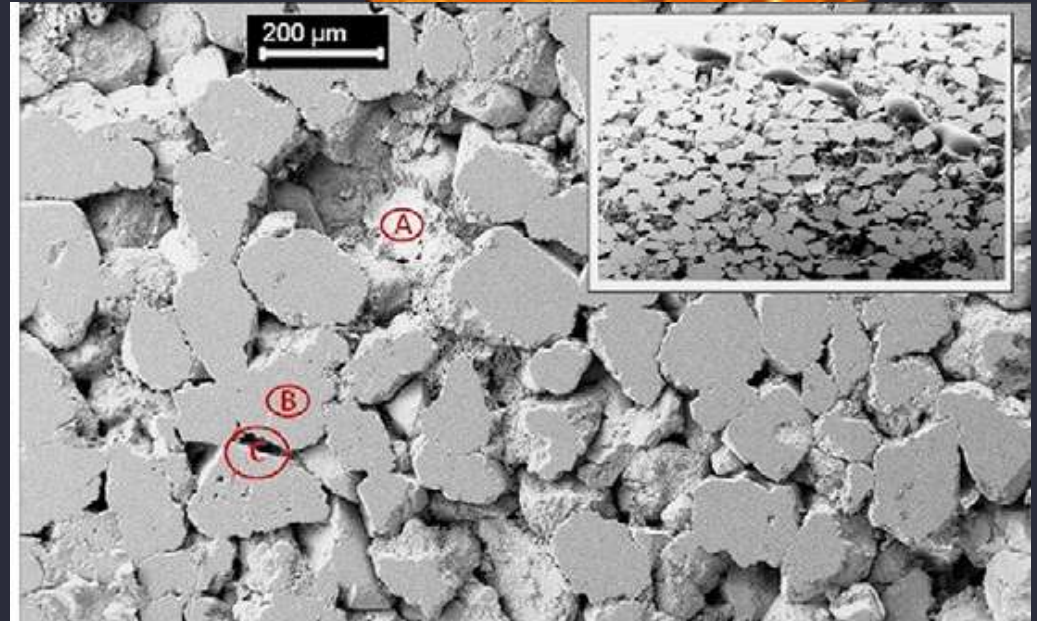
University of Alberta

Bera *et al.* 2011. *Micron*.



Characterizing the pore space in sandstone

- MicroCT
 - 3 μm in-plane, 6 μm slice-thickness
 - Bulk pores (150-200 μm)
 - Small pores (30-80 μm)
 - Suspicious micro-pores ($\sim 5\mu\text{m}$) near the detection limits
- FIB-SEM



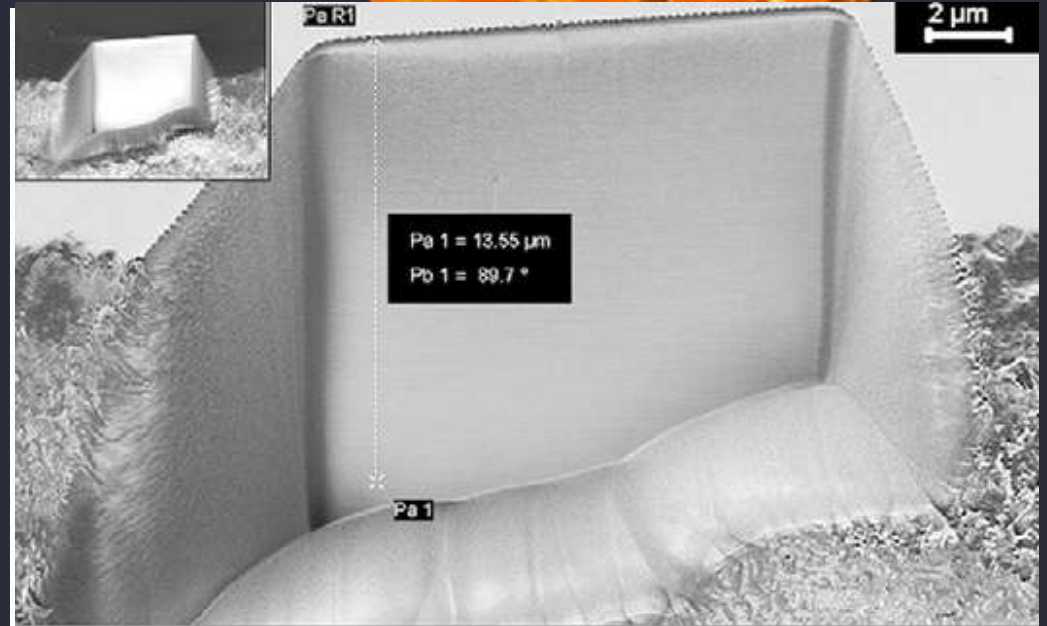
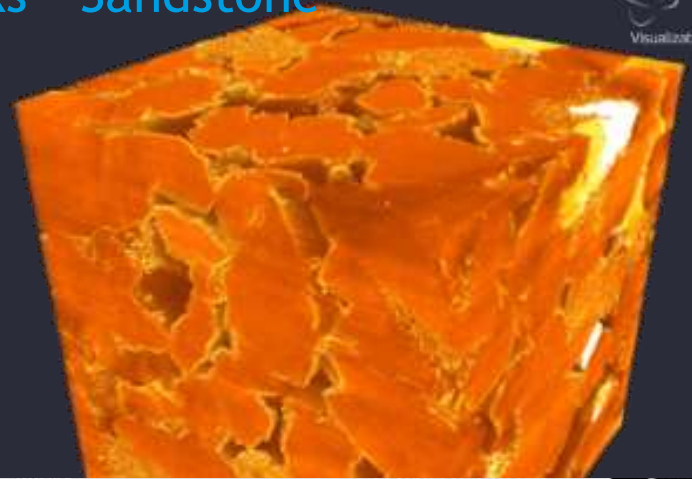
Validation of microCT porosity in berea sandstone

University of Alberta

Bera *et al.* 2011. *Micron*.

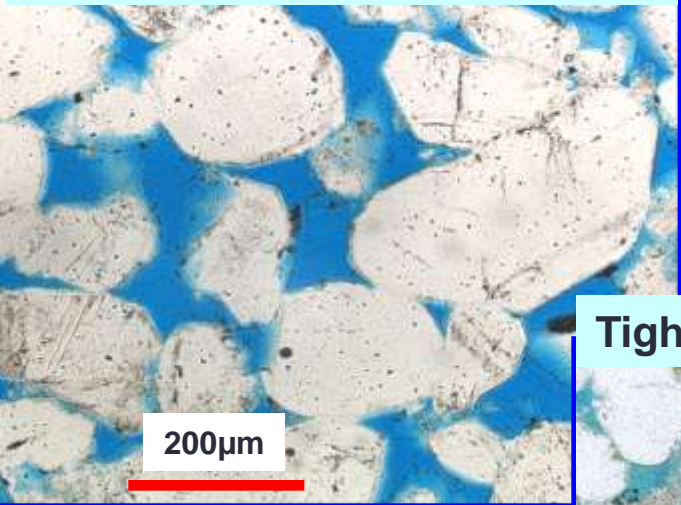
Characterizing the pore space in sandstone

- MicroCT
 - 3 μm in-plane, 6 μm slice-thickness
 - Bulk pores (150-200 μm)
 - Small pores (30-80 μm)
 - Suspicious micro-pores ($\sim 5\mu\text{m}$) near the detection limits
- FIB-SEM
 - Micro-pores are absent (artifact of x-ray microCT)



Sandstone vs. Shale reservoir rocks

Convention Sandstone Reservoir

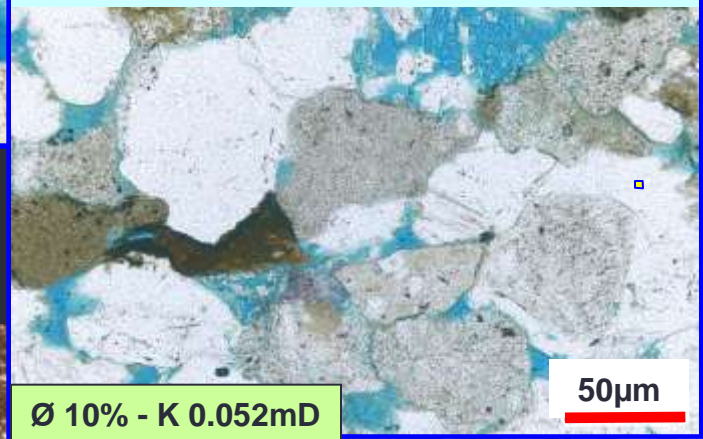


Ø 33.0% - K 753mD

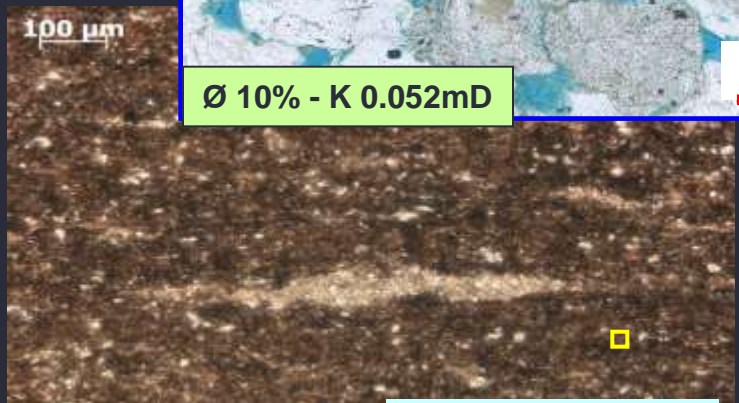


Thin section - Shale

Tight-Gas Sandstone Reservoir



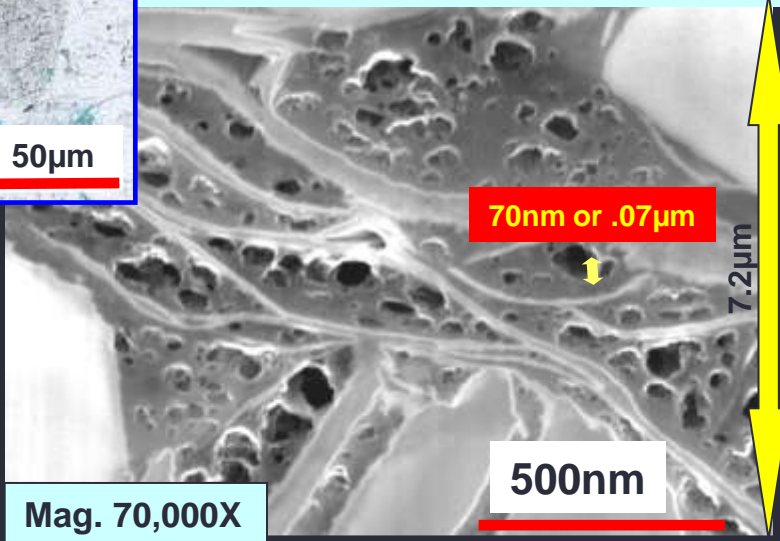
Ø 10% - K 0.052mD



Ø 2.57% K ~0.0003mD

Shale Reservoir

Shale Gas Reservoir



Mag. 70,000X

Porosity & Permeability Analysis on Nanoscale FIB-SEM 3D Imaging of Shale Rock

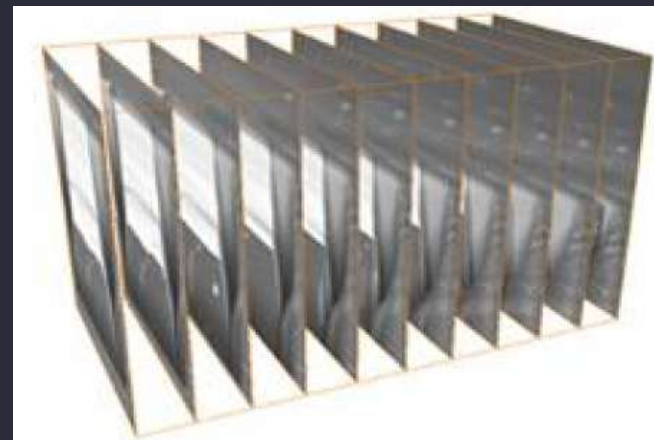
Shawn Zhang, Visualization Sciences Group - VSG

Robert E. Klimentidis, ExxonMobil Upstream Research Co.

Patrick Barthelemy, VSG

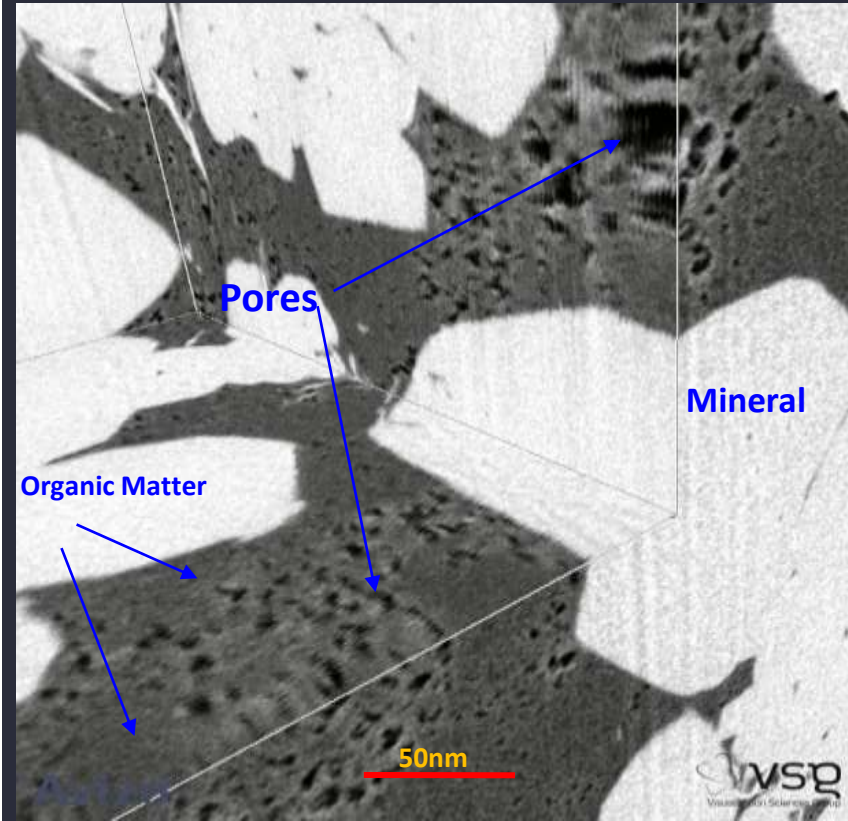
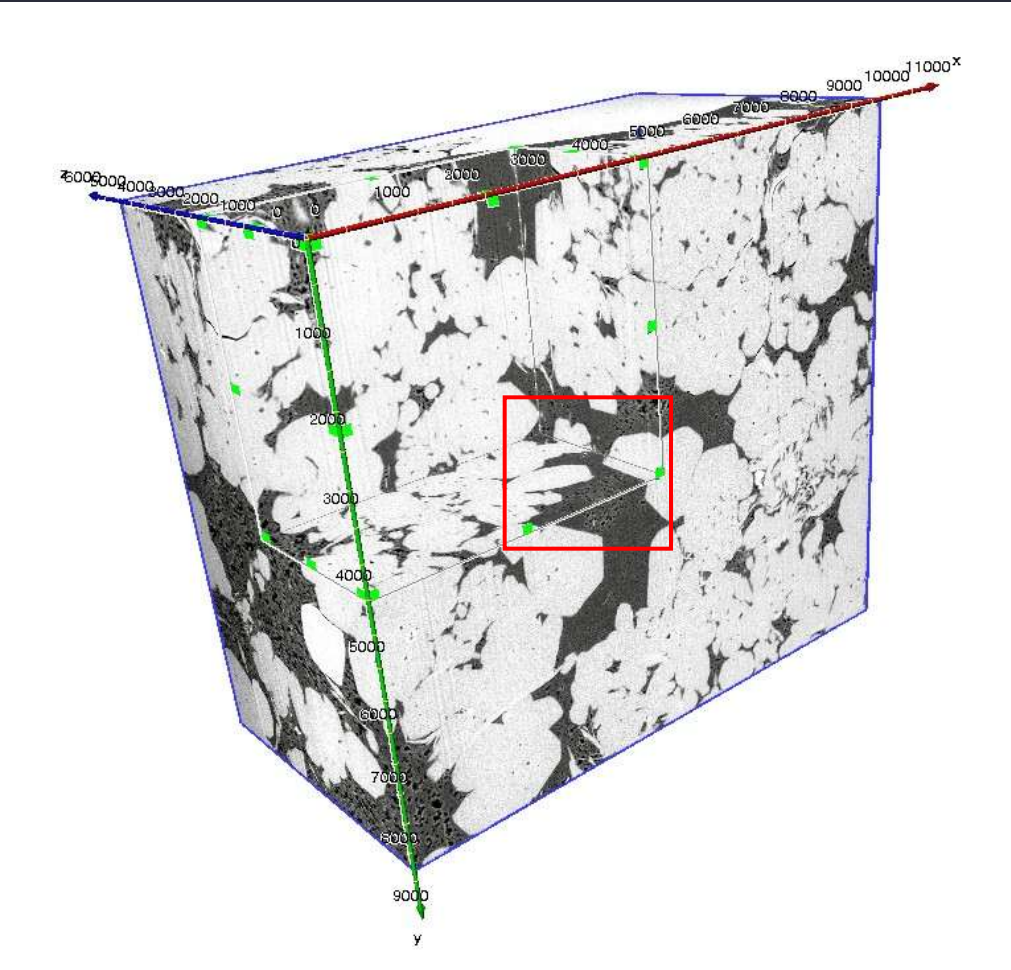
International Symposium of the Society of Core Analysts

Austin, Texas, USA, September 18th - 21st, 2011

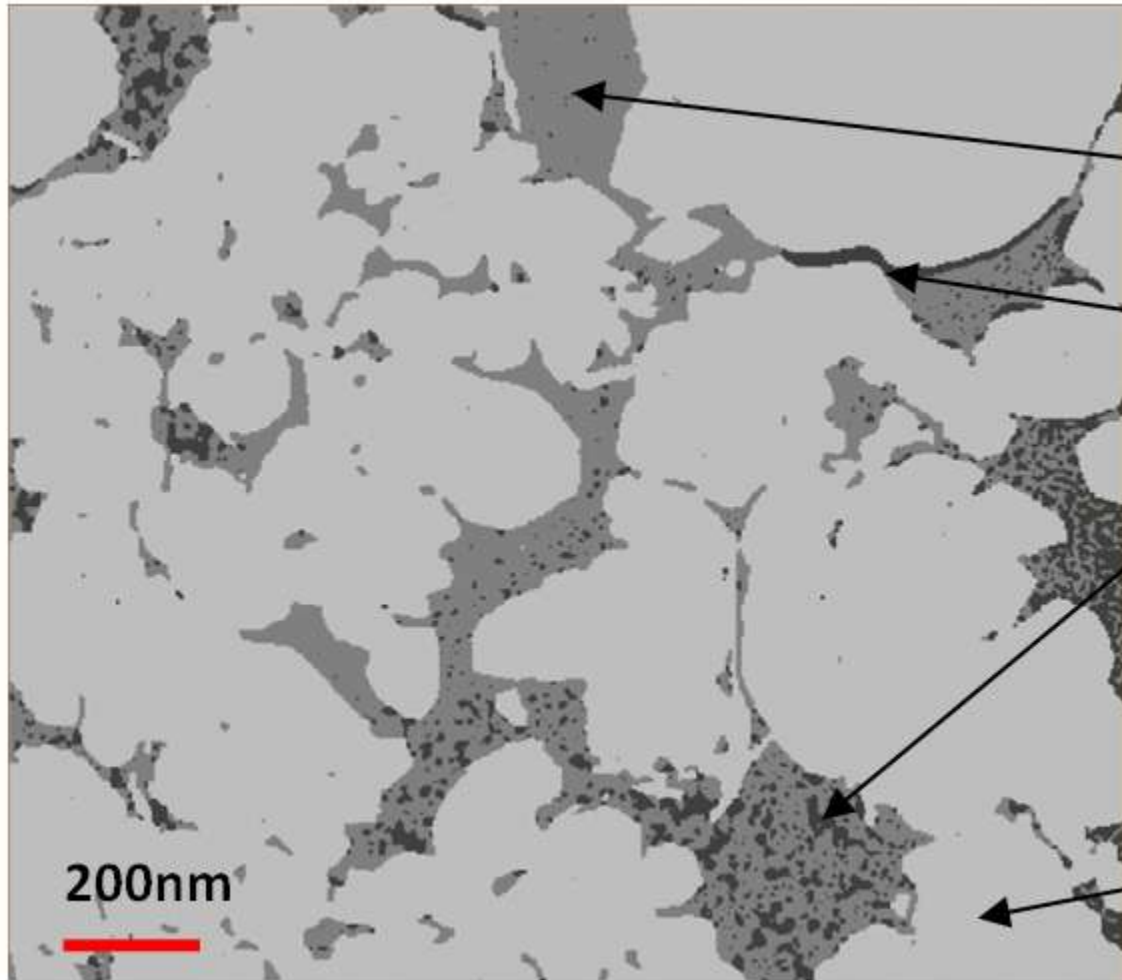


Serial slices images
5 nm voxel resolution

Volume Reconstruction



2D slice of the final segmentation results

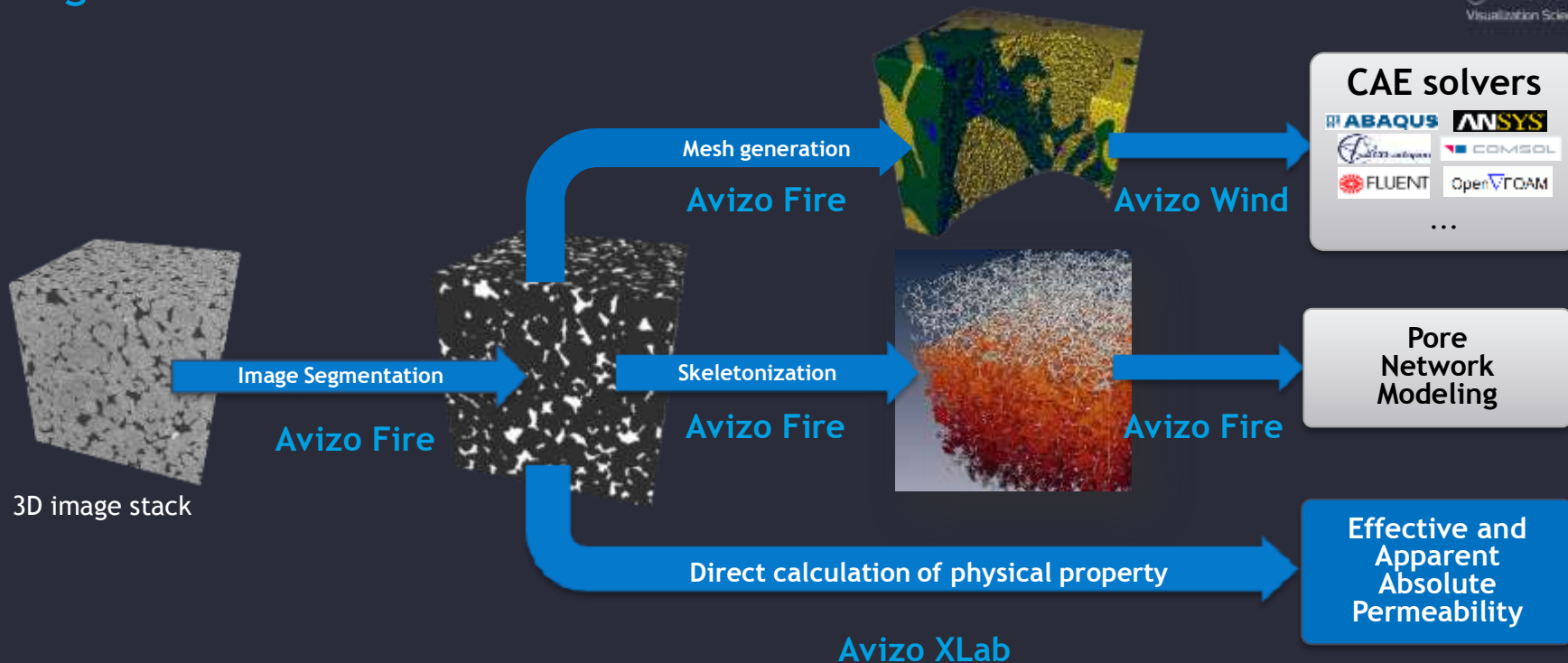


Organic Matter

Pores

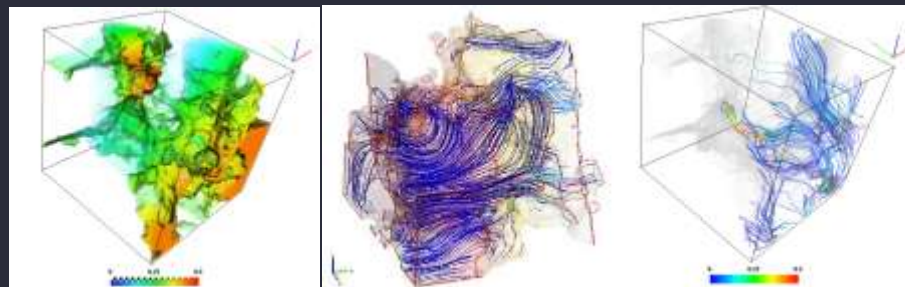
Mineral

Image-to-Simulation workflows



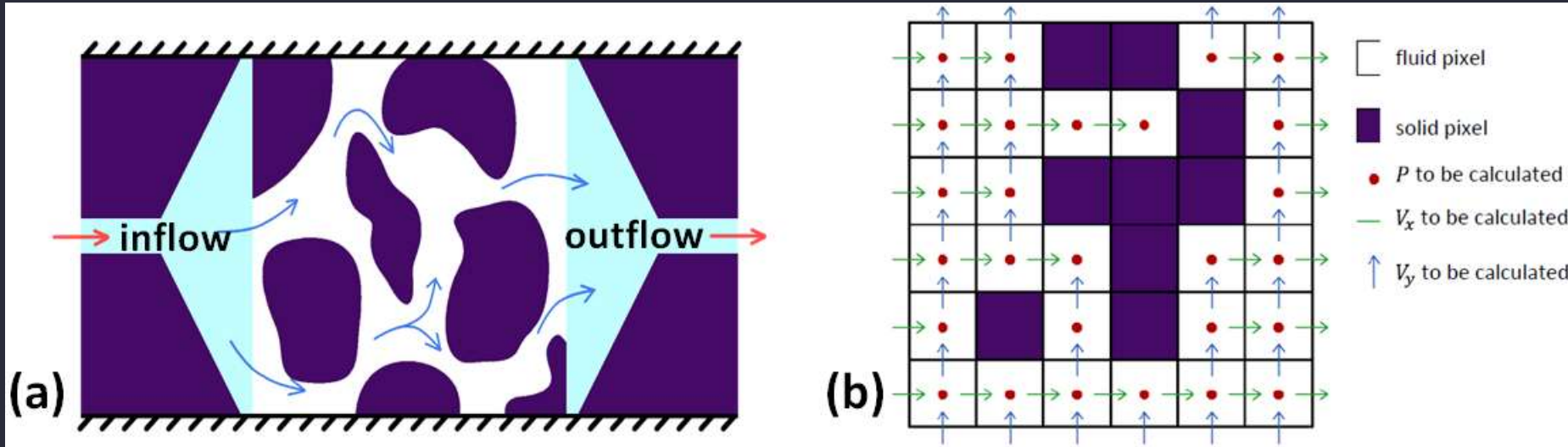
Advanced
Post-processing

Avizo Wind



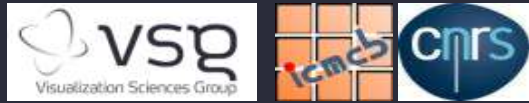
- At-a-glance,
 - Stokes flow solver
 - Finite volume scheme
 - Artificial compressibility

- Goal
 - Simplified physics & numerics
 - Much bigger problem



XLab Hydro: new extension to Avizo Fire version 7 for Materials Characterization

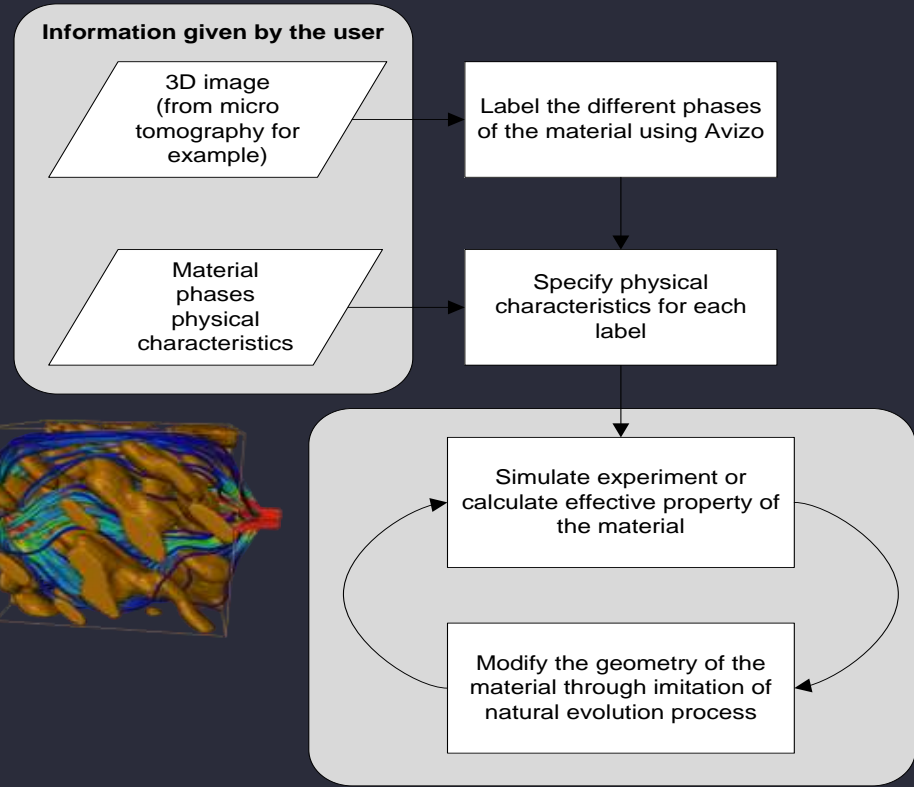
- Collaborative research



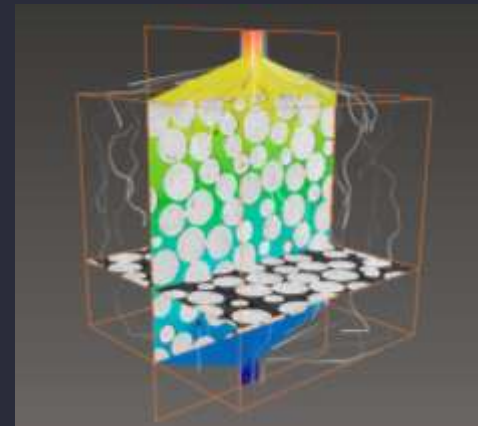
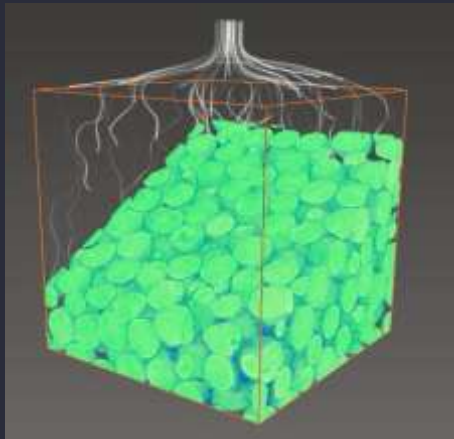
Dominique Bernard - ICMCB

- Simulator for physical properties

- Integrated with Avizo platform
- Computes:
 - Lab experiment simulation
 - Property tensor
- For **Absolute permeability (Avizo 7.0)**
- *Coming:*
 - *Heat conductivity*
 - *Electrical resistivity / formation factor*
 - *Molecular diffusivity*

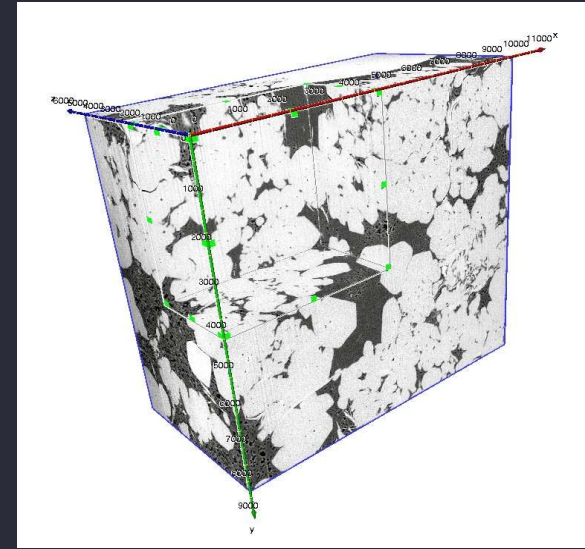


Case	Theory	Theory value	Xlab value
Square Cylinder, Side length b =25 pixel	$K=0.422b^2/12$ [7]	21.979 pixel ²	21.91 pixel ²
Round cylinder Radius r =25 pixel	$K=r^2/8$ [7]	78.125 pixel ²	78.439 pixel ²
MicroCT scan of glass packaging with spherical particles diameter d = 100-120um, material porosity $\epsilon=36.5$, 400x400x400	Kozeny-Carman equation[3] : $K = \frac{1}{180} \frac{\epsilon^3}{(1-\epsilon)^2} d^2$	6.7-9.7 um ² (6.6-9.6 D)	7.8um ² (7.7 D)



Application to shale sample - Petrophysical parameters

Volume percentage	Values
1.Total non-mineral	24.2
2.Percolating organic matter and porosity	24.1
3.Total porosity	6.25
4.Percolating porosity	5.3

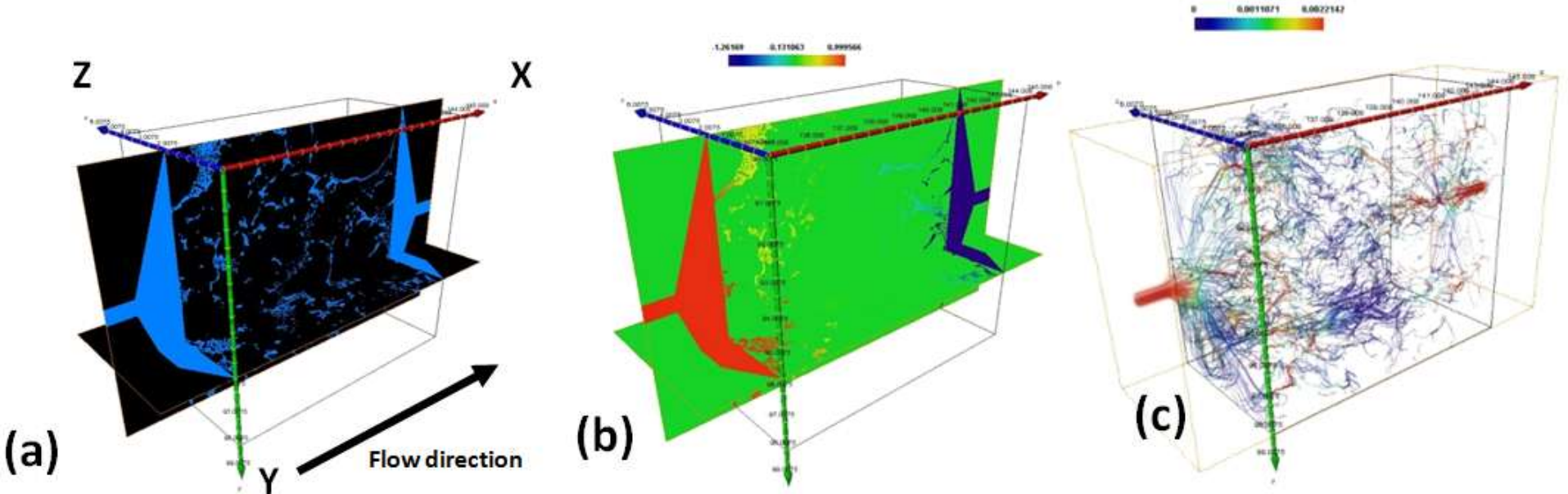


	Percolating	Total pore perm (nD)	Organic matter network perm (nD)	Shortest path (μm)	Shortest path/thickness	Longest path (μm)
X	Yes	828	156,000	11.29	1.141	14.18
Y	Yes	936	106,400	9.77	1.13	12.48
Z	Yes	280	224,400	5.84	1.08	9.49

Average = 681nD

Good agreement with experimental measures

Permeability experiment simulation using image based approach



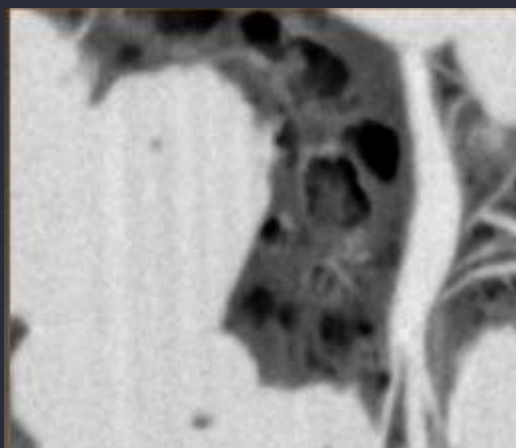
- Digital rock analysis workflow
 - Challenges in image processing and segmentation can not be under estimated
 - New solver was successful in initial validations
- More work to be done
 - Strong shadowing effect common in many FIB-SEM core sample data
 - Segmentation: Consistency, validation, and uncertainty quantification
 - Perm solver: More validation and verification
- More solvers in the works:
 - Thermal Conductivity, Winter 2011
 - Electrical Resistivity/Formation Factor, Winter 2011
 - Molecular Diffusivity, Spring 2012

Coming Avizo version 7

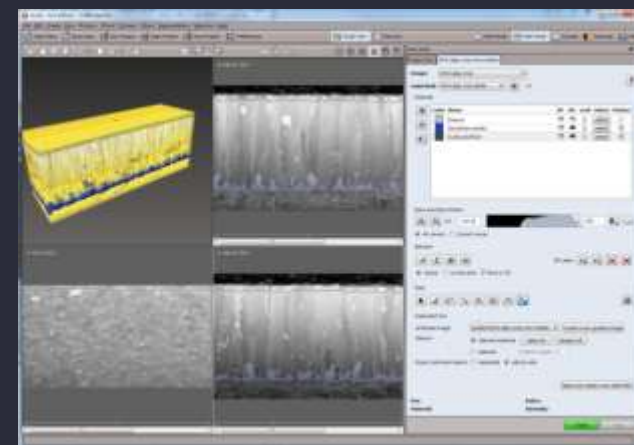
- Much enhanced and simplified user interface
- Many more image processing and analysis tools
- Enhancements for pre/post-processing numerical data
- Performance enhancements
- New extension XLab Hydro for absolute permeability



FIB Stack Wizard

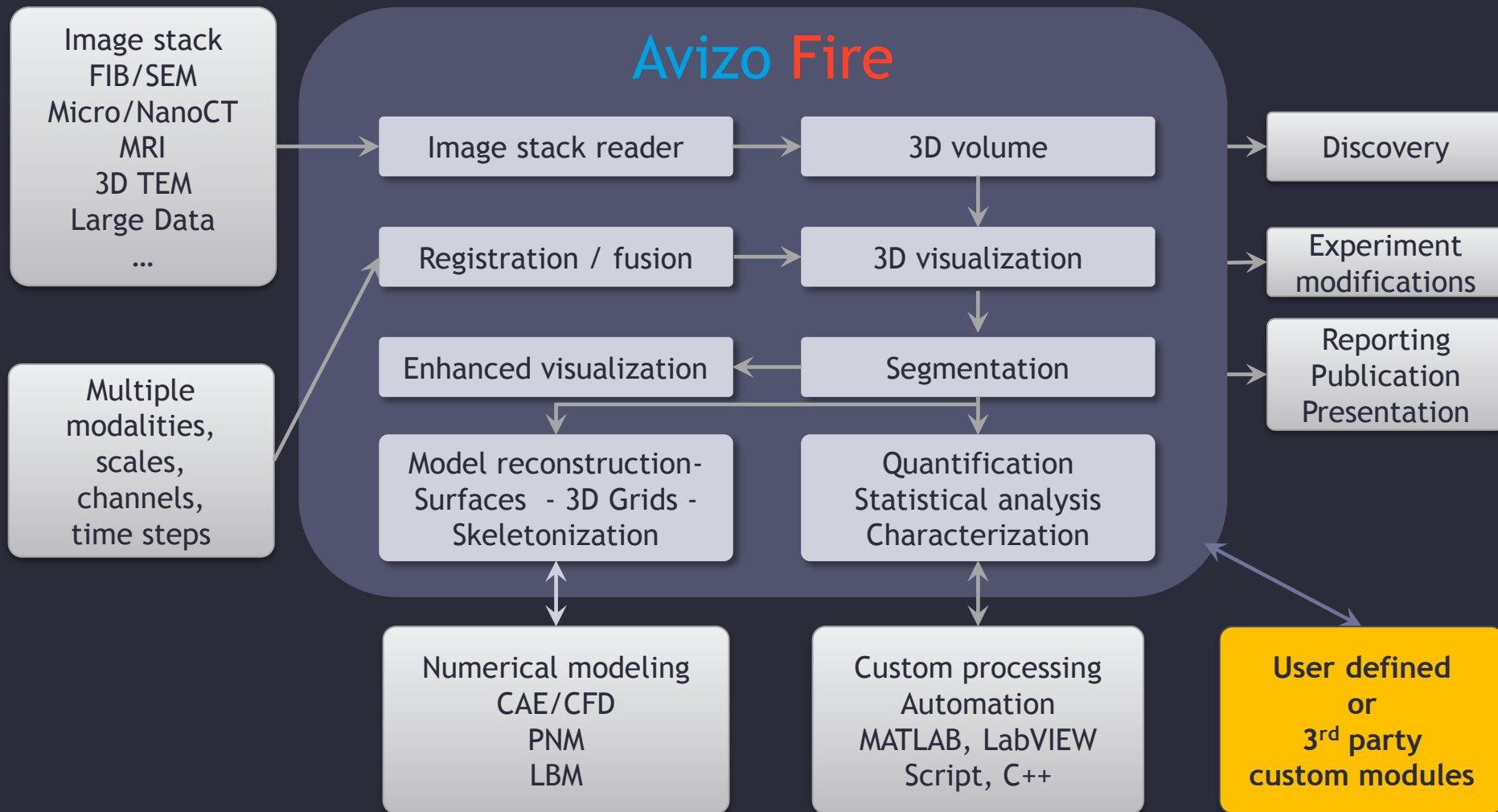


Advanced denoising filter : Non-Local Means (GPU accelerated)



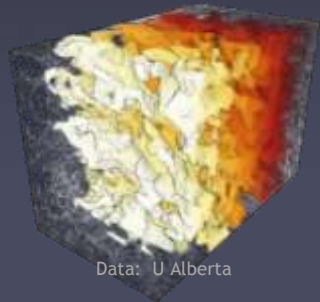
New watershed tool in segmentation editor

3D imaging and material science framework

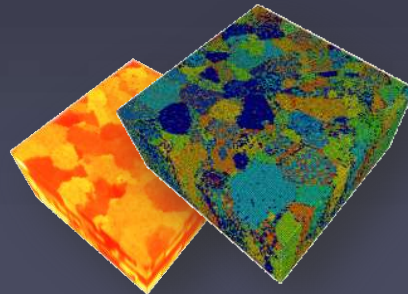
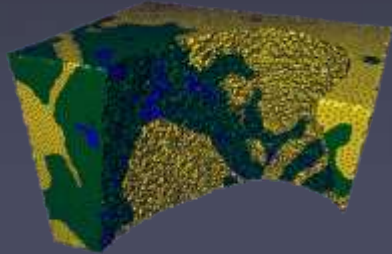




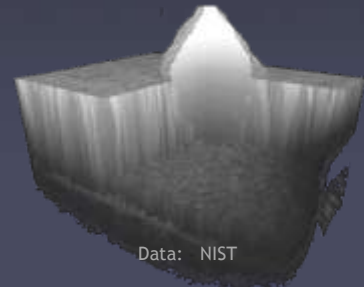
THANK YOU
daniel.lichau@vsg3d.com



Data: U Alberta



Data: U Alberta



Data: NIST